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SELECTION OF STAINLESS STEEL FOR THE  
FERMILAB ENERGY DOUBLER/SAVER MAGNETS

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Abstract

A review has been made of the choice of stainless steel to be used in the Energy Doubler/Saver magnets. The magnetic and mechanical properties of six candidate steels (304, 316, 304N, Nitronic 33, and Nitronic 40) have been evaluated. Non-ferromagnetic steels can be obtained in all grades, if the chemical composition is controlled. There are concerns about the low temperature fracture toughness of both Nitronic 33 and 40 and it is concluded that the best choice of steel for the project is either a 304N or Nitronic 40 with the nitrogen content limited to half the permitted maximum.

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SELECTION OF STAINLESS STEEL FOR THE ENERGY DOUBLER/SAVER MAGNETSI. Introduction

Austenitic stainless steel is to be used for several components in the magnets of the Fermilab Energy Doubler/Saver project. By far the major use is for the force supporting collars which resist the magnetic forces on the conductors. Selection of the stainless steel is dominated by three considerations: one, that the intrinsic magnetic moment be sufficiently low not to introduce significant harmonic errors in the magnetic field and that no significant increase in magnetic moment occur during the magnets' service life; two, that the mechanical strength and toughness be sufficient to resist the fatigue loading imposed by magnet pulsing over the service life; and three, that the steel chosen be available commercially in the required forms at a reasonable price.

The original material choice for this purpose was Nitronic 33, an Armco steel high in N and Mn and low in Ni ( $\sim 18\text{Cr } 3\text{Ni } 13\text{Mn } 0.3\text{N}$ ). Recently, low temperature testing, both at NASA<sup>(1)</sup> and NBS<sup>(2)</sup> has revealed evidence of a sharp fall off in fracture toughness at 4 K. Tests run at Fermilab<sup>(3)</sup> have shown diminished fatigue resistance compared to 316 stainless steel in measurements made at 77 K. At the beginning of this survey the principal area of concern was over the magnetic properties of Nitronic 33. The low fracture toughness properties reported have added an additional concern.

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The present report compares the properties of 6 stainless steels falling into three groups all suitable for use in the Doubler/Saver magnets.

- (i) Conventional Fe-Cr-Ni stainless steels -- types 304 and 316,
- (ii) High nitrogen strengthened Fe-Cr-Ni steels -- types 304N and 316N,
- (iii) High nitrogen, high manganese steels -- Nitronic 33 (18 Cr 3Ni 13Mn) and Nitronic 40 (21Cr 6Ni 9Mn).

The permitted composition limits of these steels are given in Table 1.

Table 1. Compositions of Austenitic stainless steels

	C	N	Cr	Ni	Mn	Si	Mo
304	.08max	see below	18-20	8-12	2 max	1 max	-
304N	.08max	0.1 - 0.2	18-20	8-12	2 max	1 max	-
316	.08max	see below	16-18	10-14	"	"	2-3
316N	.08max	0.1 - 0.2	16-18	10-14	"	"	2-3
Nitronic 33	.08max	0.2 - 0.4	17-19	2.25-3.75	11.5-14.5	"	-
Nitronic 40	.08max	0.15-0.4	19-21.5	5.5 -7.5	8 -10	"	-

- Notes:
- (i) N is generally present in 304 and 316 to ~0.03 w/o.
  - (ii) C is unlikely to be less than 0.03 w/o.
  - (iii) Mn is unlikely to fall below 0.5 w/o in 304(N) and 316(N).
  - (iv) Si is unlikely to fall below 0.25 w/o.
  - (v) These lower limits are used in calculated Ni and Cr equivalents and  $M_s$  temperatures for Table 3 and Figure 1.

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## II. Magnetic Properties of Austenitic Stainless Steels at Low Temperatures

There are three aspects to consider in determining the total magnetic moment of a stainless steel:

- (i) The paramagnetic moment of the austenite.
- (ii) The possible presence of ferromagnetic  $\delta$ -ferrite.
- (iii) The possible presence of ferromagnetic  $\alpha'$  martensite formed spontaneously on cooling below about  $\sim 200$  K<sup>(4)</sup> or with the assistance of plastic work at temperatures below about 100 C.<sup>(5)</sup>

### II(a) Magnetic Properties of Austenite

At room temperature all Fe-Cr-Ni stainless steels are paramagnetic. Depending on chemical composition they show a paramagnetic-antiferromagnet transition at  $\sim 40$  K (304) or  $\sim 20$  K (316).<sup>(6,7,8)</sup> Below the transition temperature ( $T_N$  - the Néel temperature) the magnetic moment declines as spins align themselves in an antiparallel manner. Fe-Cr-Ni steels become progressively less antiferromagnetic as their alloy content increases,  $T_N$  declining and  $\chi(4.2)$  increasing as is shown in Table 2. At about 25Cr 20Ni (type 310) antiferromagnetism is suppressed, higher alloy steels having ferromagnetic austenites at 4.2 K. Substantial additions of Mn appear to shift  $T_N$  to much higher temperatures, probably to greater than 100 K and considerably reduce the magnetic moment.

From the data of Table 2 it can be seen that Nitronic 33 and 40 have a 4 K magnetic moment which is 4-6 times lower than the 304(N) and 316(N) steels.

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Table 2. 4.2 K Susceptibility of Stainless Steels

Steel	$\chi(4.2)$	$T_N$	Reference
304L	$7.2 \times 10^{-4}$	$\sim 40$ K	4
304N	$5.7 \times 10^{-4}$	$\sim 40$ K	4
316	$9.5 \times 10^{-4}$	$\sim 20$ K	11
316LN	$9.0 \times 10^{-4}$	$\sim 20$ K	4
309	$18.7 \times 10^{-4}$	$\sim 12$ K	4
310	Incipiently ferromagnetic	-	4
Nitronic 33	$1.2 \times 10^{-4}$	$> 100$ K ?	11
Nitronic 40	$1.4 \times 10^{-4}$	"	11
Kromarc 55	$3.1 \times 10^{-4}$	"	4

All steels in annealed condition

$$\chi = M/H \quad \mu = 1 + 4\pi\chi$$

Kromarc 55 is a high Mn steel of approximate composition  
 16Cr 20Ni 9 Mn : types 309 and 310 are more highly alloyed  
 Fe-Cr-Ni stainless steels, 309  $\sim$  22Cr 14Ni, 310  $\sim$  25 Cr 20Ni

## II(b) The Occurrence of $\delta$ -Ferrite

$\delta$ -ferrite is a high temperature phase of Fe and its alloys which is frequently found in quantities of  $\sim 5\%$  in weld deposits where it considerably improves the toughness of restrained welds by reducing hot tearing. It is seldom found in wrought stainless steels which are

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annealed at 1050 C, considerably below the melting point. At this temperature  $\delta$  remaining from higher temperatures converts to austenite.

The determining factor in deciding whether a steel will have  $\delta$ -ferrite is its alloying balance its thermal history. Of the elements found in the steels considered here, C, N and Ni are austenite promoters, Cr, Si and Mo being ferrite promoters. Mn has a dual effect. At less than 6 w/o it acts as an austenite promoter but at higher contents encourages ferrite formation.<sup>(9)</sup> The relative effects of the elements as austenite promoter (Ni equivalent) or ferrite promoter (Cr equivalent) are given by the following equations:<sup>(9)</sup>

$$\text{Ni equivalent} = \text{Ni} + 0.11 \text{ Mn} - 0.0086 \text{ Mn}^2 + 18.4 \text{ N} + 24.5 \text{ C} \quad (1)$$

$$\text{Cr equivalent} = \text{Cr} + 1.21 \text{ Mo} + 0.48 \text{ Si} \quad (2)$$

Figure 1 is a plot of Cr equivalent versus Ni equivalent showing the fully austenitic and partially  $\delta$ -ferrite regions. Plotted on the diagram are rectangular areas corresponding to the range possible for each steel (see footnotes to Table 1 for details of the compositions used in Equations 1 and 2). It can be seen that, depending on exact chemical composition, all steels can form some  $\delta$ -ferrite over substantial parts of their composition range. Nitronic 33 has a small part only of its range where it would be completely austenitic when cooled from the melt. Such diagrams, generically known as Schaeffler diagrams, are well developed for Fe-Cr-Ni steels but are not so well tested for the Nitronic

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steels. Brooks<sup>(10)</sup> has confirmed that Hull's<sup>(9)</sup> diagram is accurate for 21-6-9 steels (Nitronic 40). We assume it is also valid for Nitronic 33, though to the writer's knowledge no similar study has been published. The situation is similar for 304N and 316N but since the only difference from conventional 304 and 316 is the extra N, we feel that Figure 1 accurately predicts the behavior of these two steels. It is therefore believed that a nil  $\delta$ -ferrite composition range can be specified for all steels. Greatest uncertainty attaches to Nitronic 33 due to its small  $\delta$ -ferrite range and the lack of experimental verification for this steel.

At present the magnet bore tube is seam welded. After drawing to size it is given a fully softening anneal (1/2 hour at 1050 C, fast cool). Whatever  $\delta$ -ferrite is present in the original weld should disappear in the anneal. Occasionally very small quantities remain. These quantities can only be detected magnetically. Assuming a saturation magnetization of  $\sim 1.6$  Tesla for the  $\delta$ -ferrite<sup>(4)</sup> and a 4 K susceptibility of  $1.2 \times 10^{-4}$ , we see that at a field of 1 Tesla the magnetic moments of austenite and ferrite are equal when only 0.1% ferrite is present. The experiments of Price and Yamada<sup>(11)</sup> appear to suggest that  $\delta$ -ferrite can still remain in these quantities following an anneal. Whether this is a serious concern is a question to be settled by magnetic modelling. If so, a nil  $\delta$ -ferrite composition should be selected. A similar question needs to be answered with respect to the presence of say 5%  $\delta$ -ferrite in the tack weld along the collar plates.

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II(c) Low Temperature Martensite Formation

The metastable nature of many Fe-Cr-Ni steels at room temperature and below is well known and a number of experimental investigations of its cryogenic aspects have been made.<sup>(4,5,12,13)</sup> In discussing the martensitic breakdown of austenite it is helpful to define two characteristic temperatures:  $M_d$ , the temperature at which martensite may first be formed\* by plastic deformation and  $M_s$ , the temperature at which martensite first forms in the absence of plastic deformation. For the Fe-Cr-Ni steels,  $M_d$  is known to be about 300-400 K higher than  $M_s$ .<sup>(5,13)</sup>

Following an investigation of about 30 commercial steels belonging to the grades 304(L)(N) and 316(L)(N) Larbalestier and King<sup>(4,5,14)</sup> found that the compositional dependence of  $M_s$  could be represented by the equation

$$M_s (\pm 50 \text{ K}) = 1578 - 61.1 \text{ Ni} - 41.7 \text{ Cr} - 33.3 \text{ Mn} - 27.8 \text{ Si} \\ - 1667 (\text{C+N}) - 36.1 \text{ Mo} \quad (3)$$

This equation is based on the previous work of Eichelman and Hull<sup>(13)</sup> and Hammond.<sup>(15)</sup> Hull<sup>(9)</sup> has recently extended his studies to steels high in Mn and Ni as well as higher in the region covered by Fe-Cr-Ni steels:

$$M_s (\pm 48 \text{ K}) = 1755 - 58.9 \text{ Ni} - 47.2 \text{ Cr} - 53.9 \text{ Mn} - 55.6 \text{ Mo} \\ - 37.2 \text{ Si} - 3722 \text{ N} - 2389 \text{ C} \quad (4)$$

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\*Here we concern ourselves only with the ferromagnetic martensite  $\alpha'$ . There is also a paramagnetic transition phase  $\epsilon$  whose properties are indistinguishable from its parent austenite.



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Table 3 gives the values of maximum and minimum  $M_s$  when the upper and reasonable lower composition limits of Table 1 are used. Both equations are in agreement that steels 304, 316 and 304N may transform to  $\alpha'$  martensite if towards their lowest composition limits. In general, the equations are in satisfactory agreement for the steels with  $M_s$  near or above 0 K, the Hull equation giving consistently lower values as the calculated  $M_s$  goes below 0 K. Negative Kelvin  $M_s$  temperatures of course mean no spontaneous transformation to martensite. The concept is useful in assessing the relative stability of steels to plastic deformation.

Table 3. Calculated Lower and Upper Limits to  $M_s$ 

		$M_s$ from Equation 3	$M_s$ from Equation 4
304	high	-270 K	-330 K
	low	+220	+230
304N	high	-550	-880
	low	+100	0
316	high	-410	-520
	low	+110	+100
316N	high	-690	-1070
	low	-10	-130
N33	high	-750	-1660
	low	-40	-530
N40	high	-940	-1760
	low	-120	-460

Upper and lower limits are calculated using the compositions of Table 1 and are subject to a tolerance of  $\pm 50$  K. See text for discussion of discrepancies.

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The predictions of Equation 3 have been checked with the behavior of ~30 commercially produced heats of 304(L)(N) and 316(L)(N) and agreement found to be good.<sup>(4,5,14)</sup> It is therefore believed that Equation 3 may be used to specify compositions of 304, 316 and 304N that will not transform to  $\alpha'$  on cooling to 4 K. All compositions of 316 N, Nitronic 33 and Nitronic 40 should be stable whatever their composition.

Samples of all the steels discussed here have been found to be unstable when deformed plastically to a sufficient extent at 4 K. This transformation cannot yet be quantitatively predicted and should be avoided by good mechanical design and the choice of a steel with some margin of stability.

### III. Mechanical Properties

Four out of the six steels considered here are high strength steels as may be seen from their tensile properties reported in Table 4. In each case they obtain their high strength from their high N content, up to 0.2 w/o N in 304N and 316N and up to 0.4 w/o N in the two Nitronic steels. A concern with all high strength materials is whether their fracture toughness is adequate. So far as stainless steels are concerned, the ordinary Fe-Cr-Ni steels are very tough even at 4 K but their strengths are modest. Information on the fracture toughness ( $K_{IC}$ ,  $J_{IC}$ ) and fatigue crack growth of the stronger materials is now becoming

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available through the ARPA<sup>(16)</sup> and now ERDA-DMFE<sup>(17)</sup> program on structural materials as well as other sources. Data on tensile strength as well as fracture toughness is presented in Tables 4, 5 and 6.

### III(a) 304 and 316 Stainless Steel

304 and 316 have modest strengths at 4 K, a yield strength of ~600 MPa but retain a large ratio of yield stress to tensile stress with an elongation of ~30%.  $K_{IC}$  values cannot be measured directly so large is their plastic range.  $K_{IC}$  is thus inferred from  $J_{IC}$  measurements.<sup>(18)</sup> In Table 5 it can be seen that the toughness of 316 increases on going from RT to 4 K. NBS measurements of fatigue crack growth rate show that for both 304 and 316 the fatigue crack growth rate declines a little between RT and 4 K.<sup>(19)</sup>

These steels are extremely tough and have been widely used for low temperature structures.

### III(b) High Nitrogen 304 and 316

These steels have not been as widely studied in the United States as in Britain and although they are scheduled for study in the ERDA-DMFE program as yet not as much information is available on them as on the remaining steels. Their considerable toughness can be inferred from the measurements shown in Table 6.<sup>(20)</sup> Samples of 304LN and 316LN (the low C versions are not expected to differ from the standard C steels) were warm worked by rolling until extremely high

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yield strengths were obtained (higher than in annealed Nitronic 33 and 40) and smooth and notched tensile tests made. Straight tensile tests showed substantial elongations even for a yield stress exceeding 1700 MPa ( $\sim$ 250 ksi). The notched to unnotched strength ratios remained at 1.2 or greater indicating considerable toughness at the notch root. Although less data is available for this group than either of the other two groups, the evidence that does exist indicates them to be strong, tough steels.

### III(c) Nitronic 33 and 40

Montano<sup>(1)</sup> has examined the properties of Nitronic 33 down to 20 K and his results are supported by the preliminary, unpublished results of Read<sup>(2)</sup> at NBS. As can be seen from Table 4, the tensile data indicate a sharp drop in ductility at 77 and 4 K. A 4 K fracture toughness measurement, although not completely fulfilling the requirements of ASTM E-399, was also very low.<sup>(2)</sup> A high rate of fatigue crack growth in the magnet collars themselves has also been measured at 77 K in tests conducted at Fermilab.<sup>(3)</sup> These tests made on material from three separate sources indicate that considerable questions exist concerning the 4 K fracture toughness of Nitronic 33.

The data in Tables 4 and 5 indicate that Nitronic 40 is considerably tougher than Nitronic 33. A decline in elongation and fracture toughness as the temperature is decreased from RT to 4 K is

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however, observed. Fatigue crack growth rate tests<sup>(21)</sup> have also showed a marked increase in fatigue crack growth rate at 4 K in contrast to the results already cited for 304 and 316. A considerably increased 4 K elongation of 50% has been measured by Westinghouse<sup>(22)</sup> for samples of 21-6-9 with rather lower yield stress (900-1100 MPa vs. 1240 for the NBS samples). The origin of the difference is not clear since both materials, although differently processed, came from the same starting stock. The results demonstrate the general point that ductility and toughness tend to decline as the yield stress increases. Considerable improvements in the toughness of both Nitronic 33 and 40 may be possible if the N content is restricted.

Table 4. Tensile Data on Stainless Steels

Steel	Temperature K	Yield Stress MPa	Yield Stress ksi	Tensile Stress MPa	Tensile Stress ksi	Elong. %	Ref.
304	4	570	83	1700	247	30%	23
316	4	545	79	1330	193	50	24
304LN	4	1006	146	1747	254	61	25
316LN	4	922	134	1392	202	63	25
Nitronic 33	RT	452	66	779	113	48	1
	77	1107	161	1549	225	24	1
	20	1424	207	1788	259	6	1
	4	1540	223	1810	263	5	2
Nitronic 40	RT	352	51	703	102	61	21
	77	897	130	1476	214	43	21
	4	1241	180	1634	237	16	21

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Table 5. Fracture Toughness of Stainless Steels

Steel	Temperature	Fracture Toughness		Reference
		$K_{Ic}$ MPa $m^{-1/2}$	$J_{Ic}$ kJ/m <sup>2</sup>	
304	4 K	528		18,26
316	RT		740	18,26
	4	473	875	18,26
Nitronic 33	4	(71*)		2
Nitronic 40	RT		245	18
	4K	(182*)	26	18

\* Tests did not meet all the requirements of ASTM E-399.

Table 6. Tensile Properties of Warm Worked 304LN and 316LN

Steel	Temperature	0.2% Offset Stress		Tensile stress		Elonga- tion %	Notched to un- notched strength ratio
		MPa	ksi	MPa	ksi		
304LN	RT	922	134	1040	151	30	-
	4	1550	225	2100	305	33	1.2
316LN	RT	1040	151	1100	160	23	-
	4	1760	255	2035	295	13	1.6

Data taken from reference 20

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#### IV. Summary

- (i) The lower limit to the magnetic moment of the steel is determined by the intrinsic magnetic susceptibility. At 4 K this is four to six times lower for Nitronic 33 and 40 than the 304 or 316 steels.
- (ii) All steels may be considerably more magnetic if  $\delta$ -ferrite or  $\alpha'$  martensite is allowed to form. In either case the saturation magnetization will lie in the range 14-16 mT/% of transformation product.
- (iii) If only gross quantities of  $\delta$ -ferrite (say >1%) are of concern, these may be removed by annealing the steel. This is impractical only for the tack weld on the collar laminations. It is unclear whether annealing will always guarantee less than 0.1%  $\delta$ -ferrite. If such quantities or the larger amounts in the tack weld region are likely to be objectionable, a nil  $\delta$ -ferrite steel composition should be specified.
- (iv) All the steels considered can produce  $\delta$ -ferrite depending on the balance of their alloy chemistry. Nil  $\delta$ -ferrite steels are specified with respect to the Hull diagram by favoring austenite at the expense of ferrite promoters. The 304N and 316N steels have the largest composition range in the nil  $\delta$ -ferrite region, Nitronic 33 the smallest.
- (v) Three of the six steels (304, 316, 304N) can produce martensite spontaneously on cooling to 4 K if their alloy content falls towards their lower limits. Each has an adequate margin of stability towards the high end of their composition range.

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(vi) Samples of all steels have been reported as ferromagnetic due to martensite formation following extensive low temperature deformation. Conservative mechanical design should ensure that no stress induced  $\alpha'$  is produced.

(vii) Most experience and the best mechanical property data exists for steels 304 and 316. These are both very tough steels even at 4 K. Enough evidence exists to show that Nitronic 33 has poor fracture toughness at 4K and that it should not be used for parts subject to appreciable pulsed loading.

(Viii) Nitronic 40 also shows a fall off in toughness and an increase in fatigue crack growth at 4 K although the fracture toughness remains reasonable. Little data is available for 304N and 316N. What data is available suggests excellent toughness at 4 K.

#### V. Choice of Steel

The summary of the previous section makes it clear that no one choice of steel stands out. Since the principal uncertainties concern the mechanical properties of the four most stable, high strength, high nitrogen steels, we believe that the choice of steel should be based on a careful analysis of the mechanical, particularly fatigue, loading to be experienced.

On these grounds we rule out Nitronic 33 for the collar clamps. We believe that Nitronic 40 may also be questionable and should be fatigue tested at 4 K before selection.



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Greatest toughness is obtained from the use of 304 and 316. Since no increase in fatigue crack growth occurs on going from 77 to 4 K (in fact there is a small decrease) tests can be made quite adequately at 77 K. This is also believed to be true but not demonstrated for 304N and 316N. An important point to be made is that too high a yield stress is counterproductive since it reduces fracture toughness. The yield stress of 304 at 4 K is already high (570 MPa, 83 ksi) and will allow a working strain of 0.17% if the maximum working stress is set at 60% of the yield point.

The quantity of material required for the Doubler Project is of the order 150-200 tons, most being required for the collar laminations. The relative costs of four of the six steels considered here have recently been quoted<sup>(27)</sup> as \$1.29/lb (304), \$1.60/lb (316), \$1.26/lb (Nitronic 33) and \$1.69/lb (Nitronic 40). In Britain high N versions of 304 and 316 sell for less than 5% premium on the standard grades. Since the quantity involved is sufficient to allow specification of an individual Fermilab alloy composition, we believe that each of the following choices would be satisfactory both in terms of freedom from  $\delta$ -ferrite and  $\alpha'$  martensite and in terms of adequate fracture toughness and fatigue crack growth rate at 4 K.

(i) A 304 stainless steel melted with a composition at the high alloy end of the range, balanced so as to be in the nil  $\delta$ -ferrite range. Considerable additional stability with minimum loss of fracture toughness would be obtained by increasing the Ni content above 12% to say 14%

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and/or specifying a N content of 0.1%. Similar considerations apply to 316 but since 316 is generally ~30% more expensive than 304 we believe that it is better to use a modified 304 than 316. The steel preferred is thus a low N 304N.

(ii) Nitronic 40 has considerable stability and more than adequate strength. It is believed that the toughness can be improved by reducing the N content since this is the principal determinant of the yield stress. We propose a Nitronic 40 composition melted so as to be nil  $\delta$ -ferrite with 0.2 N maximum.

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